Method of Mn in Hair Analyses Provided by Graziano's Columbia Laboratory 6.27.13

Hair collection, washing and digestion are done via a combination of two published methods,Chen et al and Das et al. To remove external surface contamination we place the hair in a beaker containing 1% Triton-X-100, sonicate for 20 minutes, rinse with deionized water (DIW) several times, add acetone and sonicate for 10 min, rinse with DIW several times and sonicate for an additional 10 min in the DIW, rinse and dried overnight at 60°C. When dry, they are placed in a acid washed 15ml polypropylene tube and weighed. One ml of concentrated HNO_3 is added to samples weighing # 100 mg and 2.5 ml is added to samples > 100 mg. Samples are left to digest for 36 hours, or until the digestion is complete. After digestion, samples are transferred into 10ml volumetric flasks (for those # 100 mg) or 25 ml volumetric flasks (for those > 100 mg) and diluted with DIW. The final acid concentration for all samples is 10%, and calibration standards for ICP-MS-DRC work are made in 10% HNO_3 .

Digested hair samples were analyzed for Mn concentration using a Perkin-Elmer Elan DRC II ICP-MS equipped with an AS 93+ autosampler. ICP-MS-DRC method for metals in hair was developed from published methods [1], with modifications and adjustments based on suggestions from Perkin Elmer application laboratory. A standard solution was used for instrument calibration. The Mn concentration of that solution was chosen to cover the expected range of metals concentrations in the hair samples: 0.2,1, and 5 ug/L. Matrix-induced interferences were corrected by selection of internal standard matched to the mass and ionization properties of the analyte. For Mn we used gallium (Ga). Stock internal standards spiking solution was prepared and added to all calibrators and samples in the same concentration, 50 ng per tube. Polyatomic interferences were suppressed with the instrument's Dynamic Reaction Cell (DRC) technology feature, utilizing ammonia as a second gas. After the instrument was calibrated, quality control samples (hair samples with known Mn concentrations obtained from the Laboratory for ICP-MS Comparison Program in Quebec) were run. Quality-control hair samples were

purchased to cover the range of concentrations of metals of interest and were run each day.

1. Pruszkowski E, Neubauer K, and Thomas R. **An Overview of Clinical Applications by Inductively Coupled Plasma Mass Spectrometry**. Atomic Spectroscopy 1998;19(4):111-115.

2. Chen, KLB, Amarasiriwardena, CJ, Christiani, DC: Determination of total arsenic concentrations in nails by inductively coupled plasma mass spectrometry. Biological Trace Element Research 67:109-125, 1999.

3. Das, D, Chatterjee, A, Mandal, BK , Samanta, G, Chakraborti, D: Arsenic in ground water in six districts of West Bengal, India: the biggest arsenic calamity in the world. Analyst 120: 917-924, 1995.